Temperature dependent defects evolution and hardening of tungsten induced by 200 keV He-ions

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Abstract

Tungsten has been selected as one of the potential candidate materials to cover some parts of the divertor in the future International Thermonuclear Experimental Reactor (ITER). The accumulation of defects and He induced by neutron irradiation and their impact on the mechanical properties of tungsten are of very importance. In this work, the high pure polycrystalline tungsten samples were implanted by 200 keV He^+ with a fluence of 5×10^{16} He^+/cm^2 at temperatures of room temperature(RT), 200, 400 and 800°C. Vacancy-type defects were detected in all implanted samples by means of positron annihilation spectroscopy. Vacancy-type defects produced by He implantation exist in the damaged layer and are decorated by He atoms. With increasing implantation temperature, more He atoms fill in the vacancy-type defects and make for the formation of larger defects. The nano-hardness values were measured by nano-indentation technique. It is observed that implantation hardening occurred for all the implanted samples. With increasing implantation temperature from 200 to 800 °C, the change of the average hardness values which are lower than the value at RT has a tendency of enhancement for the shallower layer and degradation for the deeper layer. The hardness variations are discussed to be the pinning effects of the defects with different density or size.

Keywords: positron annihilation spectroscopy, nano-indentation, He-ions implantation

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1. Introduction

Tungsten, due to its intrinsic physical properties such as high melting point, low sputtering yield with light elements and good thermo-mechanical behavior, etc., has been selected as one of the potential candidate materials to cover some parts of the divertor in ITER. The divertor, as a plasma-facing-component will be subjected to a severe environment, e.g. very high heat flux deposition, intense 14 MeV neutron, He and hydrogen isotopes bombardment at high temperature (up to ~1000 °C)[1, 2]. Neutron irradiation will cause the continuous production of He by (n,α) nuclear reactions and irradiation-induced defects. Previous studies on He atoms in tungsten mainly focused on the vacancy-like defects formation and their evolution with annealing temperature[1-6], complex defects formation[7, 8], surface morphology modification[9-13] with different fluence, temperature and energy, He retention after implantation[2, 8, 14, 15] and He thermal desorption[10, 11, 16, 17]. It is well-known that the combined presence of gas and defects often leads to microstructural and morphological modifications in the materials, such as bubbles formation and blistering, which in turn will cause the change of physical and mechanical properties [1, 18]. Despite the great interest in the mechanical properties of tungsten, few studies on the mechanical properties induced by He implantation have been performed.

Many factors including incident ion energy, incident angle, ion flux, fluence, implantation and annealing temperature have influence on the defect formation and mechanical properties of tungsten. In the present work, tungsten samples were implanted with He ions at different implantation temperatures. The formation of vacancy-like defects and micromechanical properties were investigated by Positron Annihilation Spectroscopy (PAS) and nano-indentation technology (NIT).

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2. Experimental

2.1 Samples preparation

The samples used in this study were cut from a powder-metallurgy tungsten (PM-W) plate, which was annealed at 1400 ^{o}C for an hour in the ambient of hydrogen. The purity of sample was above 99.99% and containing impurities such as 12.4148 wppm Mo、4.1651 wppm Fe、2.492 wppm Cr and 0.9659 wppm Ni etc. The size of samples was about 10×10×3.3mm^3. The samples were mechanically polished until the diamond paste with 0.25um grain, and then ultrasonic cleaned in acetone, ethanol and high-purity water in turn.

2.2 He ions implantation

Experiments of He^+ implantation were performed at 320 keV multi-discipline research platform for Highly Charged Ions equipped with an ECR (Electron Cyclotron Resonance) ion source in the Institute of Modern Physics, Chinese Academy of Sciences (IMP, CAS), Lanzhou. The beam was swept at two directions of X and Y in order to get a uniform beam. The samples were implanted with an energy of 200 keV and a fluence of 5×10^{16} ions/cm^2 and the mean flux was about 16 uA (~ 3×10^{13} ions/(cm^2 s)). The implantation temperatures were RT, 200, 400 and 800 ^{o}C. The theoretical results of the displacement damage (dpa, displacement per atom) and He concentration produced by 200 keV He in pure tungsten with a fluence of 5×10^{16} ions/cm^2 are calculated with SRIM (The Stopping and Range of Ions in Matter)-2008[19] and presented in Fig.1(a), taking an atom displacement threshold energy of 90 eV [2]. As shown in Fig.1 (a), the whole damaged region is about 600 nm from surface, the maximum He concentration at a depth of ~ 408 nm is about 3 at.% and the maximum damage at a depth of about 335 nm is about 0.5 dpa. SRIM code does not take into account any recombination process, therefore these values must be considered as the maximum possible values. Besides, the depth profile of the ratio between the calculated He concentration and dpa is given in Fig.1 (b).

2.3 Positron annihilation spectroscopy: Doppler broadening spectroscopy (DBS) measurement

Samples were probed by positrons in order to get the information about defects distribution at near surface. Measurements of DBS were carried out in Beijing ^{22}Na slow positron beam line[20]. The DBS measurement system is a standard γ-spectroscopy system equipped with a high purity Ge detector. Each spectrum was collected at RT with about 4×10^5 counts in total for each value of E and characterized by the S and W parameters. A line-shape S parameter was defined as the ratio of the area calculated around the central low momentum part of the spectrum (511±0.76 keV) over the total number of annihilation and W parameter was defined as the ratio of the area calculated in a high-momentum region far from the center of the spectrum (from 511±2.6 keV to 511±6.8 keV) over the total number of annihilation. By varying the incident positrons energy, the depth dependence of S and W can be obtained. In this work, the S(E) and W(E) were recorded in the range of 0.18 keV – 19.18 keV corresponding an incident depth $\bar{Z}$ from 0.13 to 186.40 nm. The relation of E and $\bar{Z}$ is given as follows[21]: $\bar{Z}=40E^{1.6}/\rho$, where $\bar{Z}$ is in nm，E is the energy of incident positron in keV, ρ is the density of the material in g/cm^3, (for tungsten, it is about 19.37 g/cm^3). The full-width-at-half-maximum of the implanted positrons distribution increases with energy, to reach ~300 nm at 19 keV. At this energy, positron probed up to ~500 nm under the tungsten surface and did not exceed the whole damaged region.

The S parameter is generally known to be most sensitive to the vacancy type defect present inside the sample [3] and each material exhibits specific S value, which is a signature of the momentum electrons distribution in the lattice in the absence of vacancy defects [2]. In the presence of positron trapping at vacancy-type defects, the doppler broadening curve becomes narrower and yields a higher S parameter. On the other hand, the localized positron in a vacancy-type defect has lesser overlap with core electrons than a free positron, leading to a decrease in the core annihilation parameter W [21, 22]. The W parameter is a measure of the fraction of the annihilation with high-momentum core electrons and thus it is sensitive to the chemical composition of the atoms around the annihilation site. Both parameters can be combined in S-W spectra where the different annihilation sites can be

distinguished [22].

2.4 Nano-indentation technique

The nano-indentation measurement was carried out at RT using a diamond Berkovich indenter (triangular based pyramid) in CSM (continuous stiffness measurement) mode of the Nano-Indenter G200 produced by MTS Inc. Each sample was tested at six points and and the results presented in this paper were their average values.

3. Results and discussion

3.1 Implantation-induced defects

Fig.2 gives the curves for S-parameter versus positron's mean incident depth and S vs. W plots for the un-implanted sample and the samples implanted by 200 keV He^+ with a fluence of 5×10^{16} ions/cm^2 at RT to 800 ^oC.

For the un-implanted sample, the S parameters are larger than ~0.43, which is larger than the S in the lattice and indicates the presence of intrinsic vacancy-type defects [1]. Nonetheless, it is possible to investigate the evolution of the positron annihilation characteristics of the tungsten samples with different implantation temperatures. For all the implanted samples, S parameters are higher than that for un-implanted sample, which clearly indicates that positrons detect the presence of vacancy-type defects generated during implantation of He in tungsten whatever the implantation temperature. This accords with the SRIM simulation result and indicates that the W atoms displacements can lead to the formation of vacancy-type defects which were also found in tungsten implanted by 800 keV ^{3}He[1, 2, 6]. The vacancy-type defects include mono-vacancies, di-vacancies, vacancy clusters, vacancy loops and microvoids etc, which will enhance the S parameters. In the samples implanted at RT and 400 ^oC, the S parameter increases from the surface up to ~ 7.19 nm, which is too shallow compared with the damaged layer. Beyond the maximum value, S parameters of the implanted samples at RT to 400 ^oC decrease gradually with different slopes when the mean incident depth increases. The trend of the decline can be interpreted on the basis of the He decoration of the vacancy-type defects. According to ratio between the He concentration and dpa shown in Fig.1 (b), the estimated implantation-induced defects concentration is well higher than the He concentration, which suggests that each introduced He atom can easily meet one vacancy. The injected He atoms with keV energies are trapped in irradiation-induced vacancies because of the very low migration energy (~0.3 eV [23] or ~0.24 eV [24]) of the interstitial He in tungsten at RT and the strong attractive interaction of He with vacancies in metal [2, 5, 7, 25]. Therefore, those vacancy-type defects such as dislocation/vacancy loops, monovacancy and vacancy clusters etc are all probably decorated by He, which makes for the formation of the He-vacancy (He-V) complexes with various sizes, He_iV_j. Here, the suffixes denote the number of He atoms or vacancies. When the vacancy-type defects contain gas or compound, the formation probability of positronium effectively reduces by lowering the available positron-trap volume [26], leading to a lower S parameters. Therefore, the He atoms in He-V complexes effectively reduce the low-momentum annihilation. The S parameters drop with depth mainly because the ratio between the He concentration and dpa increases from surface to ~300 nm. The difference of the slopes may be induced by the thermal migration of the interstitial He to surface.

For the sample implanted at 200 ^oC, a minimum S parameters among those of the implanted samples occurred. When the implantation temperature increases from 200 to 800 ^oC, a rise in S parameters is observed. For the sample implanted at RT to 400 ^oC, the S-W points of each sample except few points at the very near surface are almost in the same line, which signifies that one type of defects exists in the damaged layer. However, the types of defects are different for different samples. For the sample implanted at 800 ^oC, S-parameters in the region of ~200 nm from surface are nearly the same and the S-W points show that two different types of defects created in the damaged zone. Vacancies are not mobile in tungsten at RT and 200 ^oC [1, 3, 27]. Presumably the He-V complexes are only partly filled for the sample implanted at RT. For the sample implanted at 200 ^oC, the larger diffusion coefficient of He atoms could bring on more vacancy-type defects decorated by He or more He

filling in He-V complexes, which could be the reason for the much lower S-parameter and results in less interstitial He in the damaged layer. For the sample implanted at temperatures of 400 and 800 ^{o}C, vacancies are mobile[1, 3, 27] and He bubbles nucleate and grow through absorbing mobile vacancies[7]. The S parameters increase with temperature is mainly related to the formation of the larger vacancy-type defects such as vacancy clusters or He-filled microvoids at higher temperatures, which usually accompanies with the decline of defects' density. Meanwhile, the impurities like Mo, Fe, Cr, etc contained in tungsten could not trap the vacancies at higher temperatures because of the dissociation of vacancies-impurity complexes at a lower temperature of about 250 ^{o}C [3-5]. This also has positive influence on the formation of vacancy-type defects and S parameters. At 800 ^{o}C, He bubbles form in deeper region (about 270 to 500 nm from surface) where the He concentration is larger than the critical concentration for introducing He bubbles in tungsten[7]. During the implantation at 800 ^{o}C, the He atoms could concentrate in the larger He bubbles which leads to no migration of He atom[2], and at the same time the introduced He atoms could transfer along the concentration gradient due to higher diffusion coefficient. Therefore, via He atoms diffusing to surface and He bubbles, defects in the region of upper surface may be decorated by more He and less He left in the deeper region. This is the reason for the reduction of S values in the region of the upper about 50 nm and the boost of the S values in the region of 50 nm to 200 nm compared with the S values at 400 ^{o}C.

3.2 Mechanical properties

Fig.3 gives the nano-hardness profile and integrated average nano-hardness of the un-implanted sample and the samples implanted by 200 keV He^{+} with a fluence of 5×10^{16} ions/cm^{2} at RT to 800 ^{o}C. With increasing penetration depth, the nano-hardness values of the un-implanted sample decrease gradually to a constant, indicating an indentation size effect [28] of tungsten. From Fig.3 (a), the nano-hardness values of all the implanted samples are higher than the value of the un-implanted sample, which means that implantation hardening occurs. The hardness values of the implanted samples strongly depend on the type, size and density of the defects created in the damaged layer by implantation. TEM distinguishable dislocation loops [7] and relatively smaller vacancy-like defects exist in the implantation layer of the samples implanted at different temperatures. With increasing implantation temperature, the density of dislocation decreases and the size of dislocation loops increase [7]. In the course of indenter penetrating into a sample, plastic deformation arises. The implantation layer of the sample will obstacle the expansion of plastic deformation region and thus increases the strain in that region [29]. Hardness will be enhanced due to strain hardening in the plastic region [29]. Meanwhile, dislocation loops and small-volume defects as pinning points impede the motion of dislocation lines and increase the hardness [30-32]. Those are the reasons for hardening of the implanted samples.

For all the nano-hardness curves of implanted samples, there exist peaks and the depths for the peak values are not the same but all shallower than the depth for the maximum dpa and He concentration, which is consistent with previously reported results in metal[29, 33]. According to the cross section TEM image of indentation in ion implanted Fe [29], hardness profile results from the plastic deformation behavior of the implanted layer and the depth of the peak hardness corresponds to the onset of the deformation of the implanted layer. Measured values of hardness are essentially hardness values integration in an influence zone, inherent to indentation measurements [33]. Therefore, we divide the whole damaged layer (the upper 770 nm from surface) into two layers, layer I (the upper 107 nm) and layer II (107 to 700nm from surface), for the reason that the trend of hardness change in the two layers is obviously different. We suggest that the change of the integrated average nano-hardness values in these two layers could reflect the change of the micro-mechanical properties quantitatively. The average nano-hardness values for different layers of the implanted samples are concluded in Fig.3 (b). The average nano-hardness value of the un-implanted sample over the whole region is about 6.4 Gpa, which is not shown in Fig.3 (b). The maximum average hardness value is the value at RT and about 2.5 GPa greater

than the value of the un-implanted sample. With increasing implantation temperature from 200 to 800 ^{o}C, the average hardness values in layer I increase while in layer II and the whole damaged layer decrease nonlinearly. When the implantation temperature increases from 200 to 800 ^{o}C, as shown in Fig.2 (a), the S parameters increase which indicates the size of He-V complexes increases. The larger He-V complexes make for the larger stress in the surrounding materials. Therefore the expansion of plastic deformation zone becomes difficult and then the measured nano-hardness values in layer I becomes larger with temperature. The average hardness value of layer I at 800 ^{o}C is still lower than the value at RT and the average hardness values in layer II decrease with implantation temperature. This means that other factors decide the hardness at higher temperatures. We suggest that the factor is mainly the density of defects containing dislocation loops, He-V complexes and other low order defects. When the implantation temperature increases from RT to 200 ^{o}C, the reduction of interstitial He concentration and dislocation loops' density weaken the effect of point or complex defects pinning the moving dislocation. This causes the significant decline of the average hardness. For the sample implanted at 400 and 800 ^{o}C, the decrease of hardness values may be explained by the reduction of the density of dislocation loops and vacancy-type defects accompanied with the formation of larger vacancy-type defects shown in DBS results. Therefore, the reduction of the average hardness values in the whole damaged layer with increasing implantation temperature is a result of positive effect due to larger size defects and negative contribution related to density of defects and the latter plays a dominant role.

4. Summary

Modification of tungsten by the implantation with 200 keV He ions at temperature of RT to 800 ^{o}C was examined by means of NIT and PAS. Vacancy-type defects induced by He implantation are detected in the damaged layer and decorated by He atoms. With increasing implantation temperature, more He atoms fill in the vacancy-type defects and make for the formation of larger defects. Meanwhile, the NIT results indicate that implantation hardening occurs in the damaged region and with increasing implantation temperature the average hardness values increase for shallower layer I and decrease for deeper layer II. It seems that the formation of defects with larger size could primarily enhance the hardness in shallower layer and the reduction of defects' density plays a dominant role in deeper layer and the whole damaged layer.

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Figure captions:

Fig.1 Depth profile of (a) displacement damage and He concentration and (b) ratio between He concentration and dpa induced by 200 keV He in W with a fluence of 5×10^{16} ions/cm^2, taking a displacement threshold energy of 90 eV

Fig.2 (a) Curves for S-parameter vs. positron's mean incident depth (b) S vs. W plots for the un-implanted sample and the samples implanted by 200 keV He^+ with a fluence of 5×10^{16} ions/cm^2 at RT to 800 ^oC

Fig.3 (a) Nano-hardness profile and (b) integrated average nano-hardness of the un-implanted sample and the samples implanted by 200 keV He^+ with a fluence of 5×10^{16} ions/cm^2 at RT to 800 ^oC